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AD 210300

PROGRESS REPORT

October 16, 1947

to

OFFICE OF NAVAL RESEARCH
NAVY DEPARTMENT
CONTRACT NO. N5-ORI-111,
PROJECT ORDER NO. 1

on

"Alloys and Ceramic Materials for High-Temperature Service"

BATTELLE
MEMORIAL INSTITUTE
505 King Avenue
COLUMBUS 1, OHIO

FORM 49

BATTELLE MEMORIAL INSTITUTE
INDUSTRIAL AND SCIENTIFIC RESEARCH
COLUMBUS 1, OHIO

October 31, 1947

Chief of Naval Research
Navy Department
Washington 25, D. C.

Attention Mechanics and Materials Branch

Dear Sir:

I am enclosing four copies of the report covering the work done during the month of September, 1947, on "Alloys and Ceramic Materials for High-Temperature Service", carried out under Navy Contract No. N5-ori-111.

As directed by the Mechanics and Materials Branch, other copies of the report have been distributed as indicated by the distribution list bound with the report.

We shall be pleased to have any comments you or your associates, or any person receiving this report, may care to make with regard to the research.

Preparation of summary reports on the five phases of the work under this contract is continuing.

Sincerely,

Howard C. Cross

Howard C. Cross

HCC:ma
Enc. (4)

C-599857

PROGRESS REPORT

on

ALLOYS AND CERAMIC MATERIALS FOR HIGH-TEMPERATURE SERVICE

to

OFFICE OF NAVAL RESEARCH, NAVY DEPARTMENT
CONTRACT NO. N5-ORI-111, PROJECT ORDER NO. 1

BATTELLE MEMORIAL INSTITUTE

October 16, 1947

PROGRESS REPORT DISTRIBUTION LIST

"Alloys and Ceramic Materials for High-Temperature Service"
Battelle Memorial Institute
Contract N5-ORI-111

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PROGRESS REPORT

on

ALLOYS AND CERAMIC MATERIALS FOR HIGH-TEMPERATURE SERVICE

to

OFFICE OF NAVAL RESEARCH, NAVY DEPARTMENT
CONTRACT NO. N5-ORI-111, PROJECT ORDER NO. 1

from

BATTELLE MEMORIAL INSTITUTE

October 16, 1947

SUMMARY

This report covers the work done during September, 1947.

Four chromium-base alloy heats, of the 60Cr-15Fe-25Mo type, were tested in stress-rupture at 1600°F.

Curves of stress versus rupture time and creep rate for GT-45 alloy at 1500°F. are presented.

Curves of tensile-fatigue test data for cast and wrought alloys that have been tested to date at 1200, 1350, and 1500°F. are given.

Eight 60Cr-15Fe-25Mo type alloys were prepared in the vacuum apparatus.

The experimental work involved in checking the present working diagram for the cobalt-chromium binary system is nearing completion.

In the study of the causesof cracking in welds and adjacent parent metal, metallographic examination was continued on Timken alloy circular-groove specimens welded with a variety of electrodes and on Timken alloy-Vitallium wheel-and-bucket replicas welded with Types 316 (18Cr-12Ni-2Mo) and 349 (19-9 W-Mo) electrodes.

Changes in the physical properties of No. 38900 Alundum specimens, which were burned in the temperature range of 1000 to 3300°F., occurred at relatively uniform rates. Within the limits of this investigation, no sudden or abrupt changes were observed. In general, and as was anticipated, the modulus of rupture, bulk density, and linear shrinkage increased, and the water absorption decreased as the burning temperature was increased. Specimens burned at temperatures of 2250°F. or lower, however, were extremely fragile, and, as a result, were unsuitable for strength determinations.

A dilatometric study of a No. 38900 Alundum specimen indicated that an abrupt expansion and an equally abrupt contraction occurred over the temperature range of 55 to 175°F., and contractions over the ranges of 800 to 875°F. and 1700 to 1820°F. The expansion of the body from room temperature to 1700°F. occurred at a uniform rate, except in the ranges indicated.

The tensile strength of a specimen of No. 38900 Alundum, reburned at 3300°F., was 1500 pounds per square inch when tested at 1800°F. A very coarse crystalline structure was apparent throughout the tensile specimen.

ENGINEERING PROPERTIES OF HEAT-RESISTING ALLOYS

(Work done by C. W. Andrews, Ward F. Simmons, and Howard C. Cross)

Experimental Work

Test Materials

The chemical compositions of the alloys tested are shown in Table 167.

The creep test specimen of GT-45 alloy and the tensile-fatigue specimens of S-590 alloy were machined from bar stock. The tensile-fatigue specimens of 422-19 alloy and Vitallium were machined from oversized precision castings, since they could not be cast to size with sufficient accuracy for these tests.

The stress-rupture test specimens of the 60Cr-15Fe-25Mo type alloy were ground from chill castings which had been stress-relieved, after being removed from the mold and before cooling to room temperature, by heating to 1200°F. and furnace cooling.

The heat treatments for the alloys tested are given in the footnotes of Tables 168 and 169.

Stress-Rupture Tests

Five stress-rupture tests of chromium-base alloy specimens and a creep test of a specimen of GT-45 alloy were in progress during September, 1947. Data for all six tests are shown in the table of stress-rupture data (Table 168).

TABLE 167. CHEMICAL COMPOSITIONS OF ALLOYS TESTED

Alloy	Alloy Number	Composition, Per Cent												
		C	Mn	Si	S	Cr	Ni	Co	Mo	W	Cb	Fe	N_2	Zr
GT-45 (046012)	NR-100	0.088	1.24	0.66	-	16.79	14.19	Cu 3.01	2.75	Ti 0.28	0.42	Bal.	-	-
Vitallium (cast)	NR-10L	0.27	0.70	0.70	-	27.26	3.06	Bal.	5.31	-	-	1.40	-	-
422-19 (cast)	NR-12B	0.43	0.70	0.60	-	26.91	15.32	Bal.	6.27	-	-	1.35	-	-
S-590 (Lot No. 2)	NR-74	0.47	1.35	0.82	-	19.40	19.07	19.26	4.03	4.00	3.87	Bal.	-	-
Chromium-base	A-3457	0.16	-	0.58	-	55.1	-	-	26.8	-	-	15.9	0.050	-
Chromium-base	A-3466	0.04	Nil	0.04	0.006	63.0	-	-	22.4	-	-	13.2	0.007	-
Chromium-base	A-3831	0.06	0.04	0.70	0.005	59.8	-	-	24.0	-	-	14.9	0.027	(a)
Chromium-base	A-3832	0.08	0.07	0.57	0.003	54.0	-	-	27.6	-	-	16.2	0.030	(a)

(a) Will be reported later.

TABLE 168. STRESS-RUPTURE DATA

Material	Specimen Number	Temperature, °F.	Stress, p.s.i.	Rupture Time, Hours	Elongation, %	Reduction of Area, %	Minimum Creep Rate, % Per Hour	Hours for Deformation of 1%	2%	5%
GT-45 (046012)	NR-100-12(a)	1500	8,000	(b)	-	-	.000017(b)	-	-	-
Chromium-base	A-3457(c)	1600	45,000	50.5	0.7	1.6	.021	31	-	-
Chromium-base	A-3466-1(c)	1600	45,000	23.2	2.4	2.4	-	-	-	-
Chromium-base	A-3466-2(c)	1600	38,000	88.8	8.5	6.3	.038	23	37	70(d)
Chromium-base	A-3832(c)	1600	38,000	(Broke on loading)						
Chromium-base	A-3831(c)	1600	38,000	(Broke on loading)						

(a) 2250°F. for 1/2 hour, water quenched, aged for five hours at 1200°F., water quenched, plus five hours at 1350°F., water quenched.

(b) Creep test, 2091 hours duration. Creep rates at 500, 1000, 1500, and 2000 hours were .00003, .000017, .000065, and .00019 per cent per hour, respectively. Deformations at the same times were 0.125, 0.133, 0.154, and 0.230 per cent, respectively. Initial deformation was 0.044 per cent.

(c) 1/4-inch-diameter specimen, stress relieved by removing from the mold before cooling to room temperature, heating to 1200°F., and furnace cooling.

(d) Estimated value.

The five chromium-base alloy specimens were tested at 1600°F., three of them at 38,000 p.s.i. and two at 45,000 p.s.i. Two of the specimens which were to be tested at 38,000 p.s.i. (A-3831 and A-3832) broke on loading; the third (A-3466-2) lasted 89 hours at that stress and showed good ductility - 8.5% elongation and 6.3% reduction of area. As indicated in the section on "Chromium-Base Alloys", efforts are being made to decrease the shock-sensitivity and brittleness of the 60Cr-15Fe-25Mo type alloy. From the information obtained from a few heats, it appears that the addition of small amounts of manganese seems to reduce the sensitivity of this alloy to cracking. In continuation of this program, Heats A-3831 and A-3832 were treated with Si-Zr alloy and no manganese addition was made; one of the two test bars from each heat was rejected because of cracks. Heat A-3466 was made using the purest available melting stock and was deoxidized with carbon.

Of the two specimens loaded at 45,000 p.s.i., Specimen A-3457 lasted 50 hours and A-3466-1 23 hours. These specimens showed low ductility - 0.7 and 2.4 per cent elongation, and 1.6 and 2.4 per cent reduction of area, respectively.

The rupture data for Specimens A-3466-1 (45,000 p.s.i.) and A-3466-2 (38,000 p.s.i.) fell below the logarithmic stress versus rupture time curve for this chromium-base alloy. The data for the test of Specimen A-3457 (45,000 p.s.i.) were slightly above the curve.

Creep Tests

A creep test of GT-45 alloy (Heat 046012 , Specimen NR-100-12) was completed after 2091 hours at 1500°F. and 8,000 p.s.i. At 500, 1000, 1500, and 2,000 hours, the creep rates were .00003, .000017, .000065, and .00019 per cent per hour, respectively, and total deformations were 0.125, 0.133, 0.154, and 0.230 per cent, respectively. Transition to third-stage creep took place at about 1200 hours (0.135% total deformation). This test and previous stress-rupture tests on specimens from Heat 046012 gave creep and stress-rupture properties that were much superior to the results given in earlier reports for Heat 36282 of the same alloy. Figure 112 shows the stress versus creep rate and rupture time curves for Heat 046012 of GT-45 alloy at 1500°F.

Tensile-Fatigue Tests

The tensile-fatigue testing programs at 1350°F. have been completed on Vitallium (NR-10L) with a stress variation from the mean of ±15,000 p.s.i. and on S-590 alloy (NR-74) with a stress variation from the mean of ±25,000 p.s.i. The tensile-fatigue testing program of 422-19 alloy (NR-12B) has been suspended while load-maintaining devices are being installed on the fatigue-testing machines.

Data for all of the tests on Vitallium, 422-19 alloy, and S-590 alloy are shown in Table 169. Figure 113 shows curves for most of the cast and wrought alloys that have been tested to date at 1200, 1350 and 1500°F.

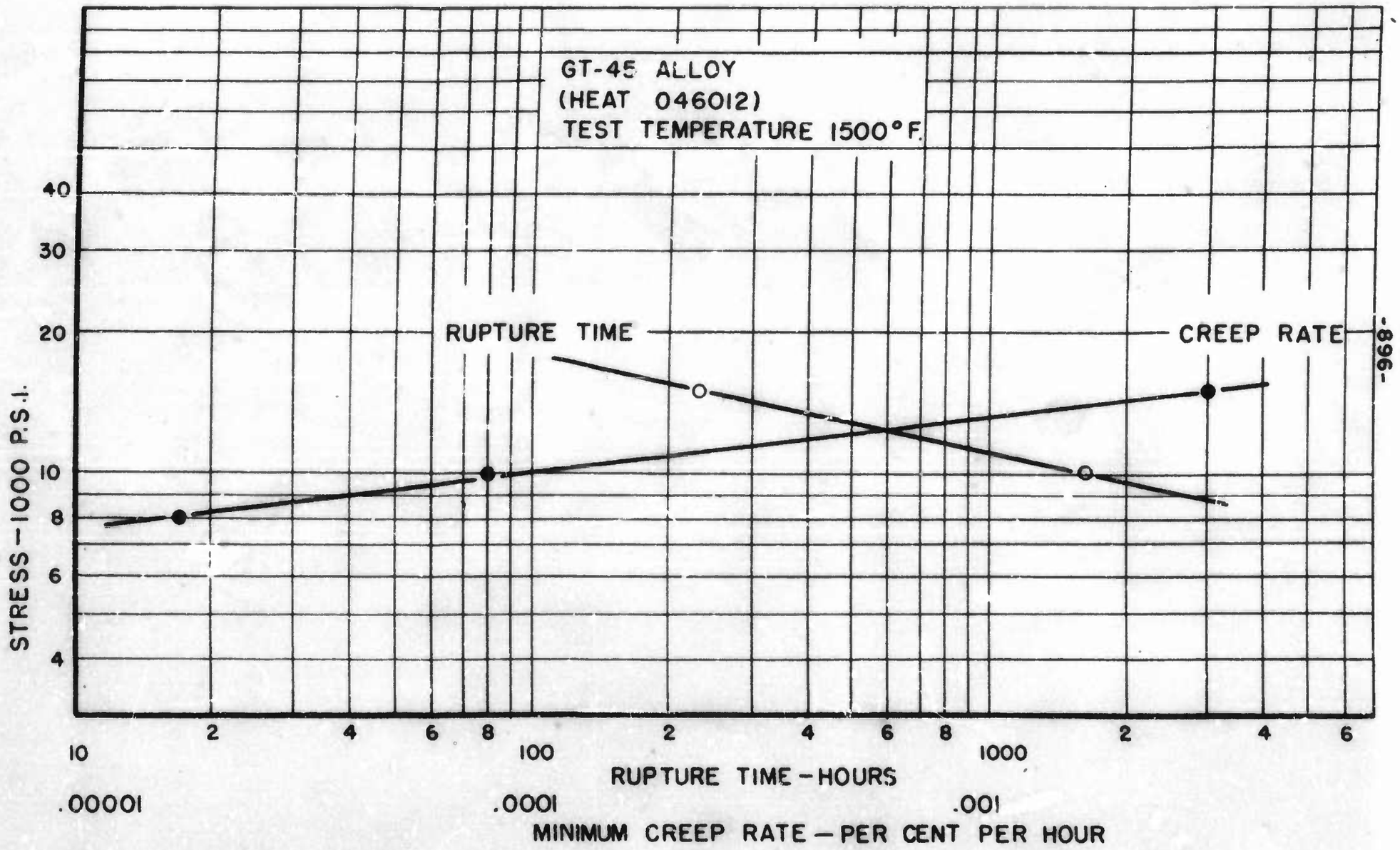

FIGURE 112. STRESS VS. RUPTURE TIME AND MINIMUM CREEP RATE FOR GT-45 ALLOY (HEAT 046012) AT 1500° F.

0-6469

TABLE 169. TENSILE-FATIGUE TEST DATA

(Stress Amplitude ±15,000 p.s.i. From the Mean Stress Unless Otherwise Indicated)

Material	Specimen Number	Temperature, °F.	Mean Stress, p.s.i.	Cycles to Failure, Millions	Time to Rupture, Hours*
Vitallium (cast)(a)(k)	NR-10L-8	1350	45,000	1.86	21
Ditto	NR-10L-4	1350	40,000	2.70	30
"	NR-10L-3	1350	35,000	12.00	133
"	NR-10L-2	1350	30,000	27.23	303
"	NR-10L-5	1350	26,000	(c)	(c)
"	NR-10L-6	1350	26,000	86.78	964
422-19 (cast)(a)	NR-12B-5	1350	50,000	0.541	6.0
Ditto	NR-12B-1	1350	45,000	4.780	53.2
"	NR-12B-3	1350	40,000	39.60	440
"	NR-12B-2	1350	37,000	(d)	(d)
"	NR-12B-6	1350	37,000	29.58(e)	329(e)
"	NR-12B-4	1350	37,000	(f)	(f)
"	NR-12B-8	1350	37,000	(g)	(g)
"	NR-12B-7	1350	37,000	33.52	373
S-590 (b)(l)	NR-74-81	1350	40,000±25,000	0.106	1.2
Ditto	NR-74-84	1350	40,000±25,000	0.113	1.3
"	NR-74-82	1350	35,000±25,000	1.119	12.4
"	NR-74-91	1350	30,000±25,000	5.220	58
"	NR-74-92	1350	26,000±25,000	(h)	(h)
"	NR-74-86(i) (j)	1350	26,000±25,000	66.97	744

(a) Aged for 50 hours at 1350°F.
(b) 2275°F. for 40 minutes, water quenched, and aged for 16 hours at 1400°F.
(c) Power failure caused a temperature drop and stress increase at 21.47 x 10^6 cycles (238 hours*); test discontinued.
(d) Power failure caused a temperature drop and stress increase at 33.86 x 10^6 cycles (376 hours*); test discontinued.
(e) Cycles and hours to failure were less than for test on NR-12B-3 at higher stress of 40,000 ± 15,000 p.s.i. A duplicate test on Specimen NR-12B-4 was therefore started.
(f) Power failure at 0.3 x 10^6 cycles. Test discontinued.
(g) Resistor in power relay failed at 6.92 x 10^6 cycles, causing a considerable temperature drop and stress increase. Test discontinued and specimen replaced with a duplicate, to be tested at the same stress.
(h) Power failure caused a temperature drop and stress increase at 26.75 x 10^6 cycles (297 hours*); test discontinued.
(i) Erroneously designated NR-74-93 in Table 143 of the report dated July 16, 1947
(j) Power failure caused a temperature drop and stress increase at 28.5 x 10^6 cycles (316 hours*). The test was continued after the specimen had been cooled and checked for visible damage.
(k) Series of tests complete at 1350°F. with stress cycle ±15,000 p.s.i.
(l) Series of tests complete at 1350°F. with stress cycle ±25,000 p.s.i.

* Note: The time in hours is the actual running time of the test, and does not include any time when the machine was not running and the load was at the mean or the minimum value.

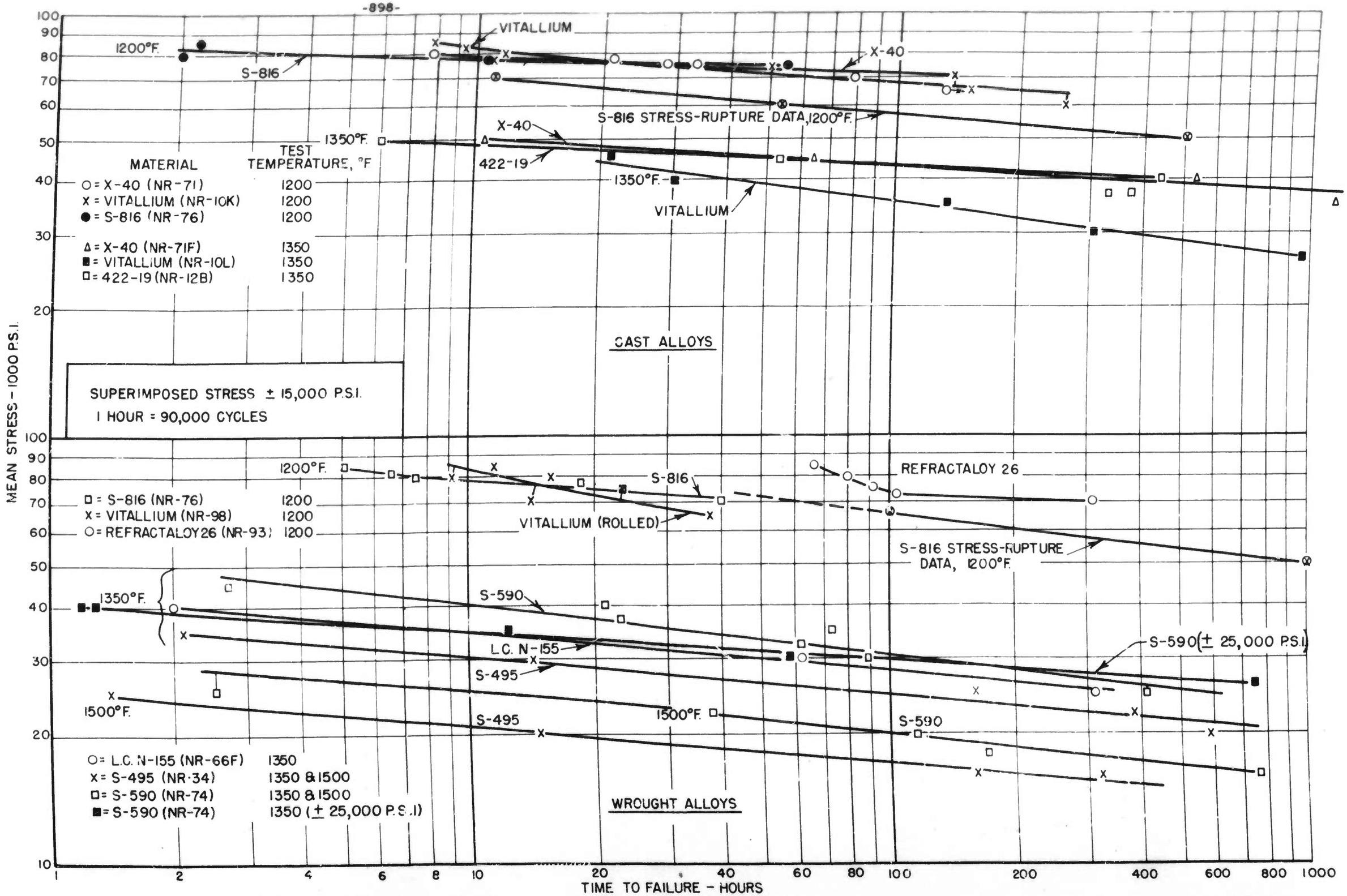

FIGURE 113. SUMMARY OF TENSILE-FATIGUE DATA AT 1200, 1350, AND 1500°F.

0-6179

The effect of the superimposed tensile-fatigue stress on the rupture life differs between the various alloys. At high mean stresses, forged S-495 and S-590 alloys, and cast 422-19 and X-40 alloys show shorter life under tensile fatigue (±15,000 p.s.i. from the mean) than under similar static tension stresses. At lower stresses and longer rupture times, the life under fatigue conditions is almost equal to, and in some cases surpasses, the life under static tension stress. Over the range of stresses used, Vitallium has shown equal or longer life under fatigue conditions.

For S-590 alloy at 1350°F. tested with a stress amplitude of ±25,000 p.s.i. from the mean stress, shorter rupture times were obtained at high stresses than for either constant stress tests or tensile-fatigue tests cycled ±15,000 p.s.i. from the mean stress. However, a test at a mean stress of 26,000 p.s.i. was superior to similar tests cycled ±15,000 p.s.i. and equal to the results obtained in static tension tests.

When the automatic load maintainers have been installed on the Krouse fatigue machines used in this work, check tests will be made to determine whether or not the idle time (when the machine was not running and the load was at the mean or the minimum value) has had any significant effect on the data obtained.

CHROMIUM-BASE ALLOYS

(Work done by W. L. Havekotte and D. E. Krause)

Introduction

The effort for the month of September was directed along the following lines:

1. Six of the previously prepared 60Cr-15Fe-25Mo type alloy heats are being analyzed chemically and examined metallographically.

2. Eight 60Cr-15Fe-25Mo type alloy heats were prepared in the vacuum equipment. Zirconium and selenium, individually and in combination with manganese, were added to these melts in an effort to improve the toughness of the chromium-iron-molybdenum alloy.

Experimental Work

Chemical Analyses and Metallographic Examination of Chromium-Base Alloy Heats Made in August

During the month of August, nine 60Cr-15Fe-25Mo type alloys were melted and cast in vacuum following the melting procedures recorded on Page 872 of the report dated September 16, 1947. Chemical and vacuum fusion analyses and grain size of six of these heats are recorded in Table 170. Three of the heats were not analyzed because the desired vacuum was not obtained during melting. The castings from these will be held for possible future use.

The chromium contents of Heats A-3460, A-3464, and A-3465 were lower than desired. The chromium content of Heat A-3466, in which chromium was added as minus 200-mesh powder, was higher than anticipated. It was expected that the use of fine particles of chromium would result in a higher chromium loss, but this was not the case.

The nitrogen content of Heat A-3466, in which the purest obtainable melting stock was used, was lower than that of any previous melt. This may have been the result of low nitrogen in the charge, or perhaps the result of a more vigorous than usual boiling of the melt caused by the high

TABLE 170. ANALYSES, GRAIN SIZE, AND MELTING DATA FOR CHROMIUM-BASE ALLOY HEATS

Heat Number	Analysis, Per Cent											ASTM Grain Size	Pressure, Microns, at		Tapping Temperature, °F.	Melting Procedure Number*
	Wet Method								Vacuum-Fusion Method							
	Cr	Fe	Mo	C	Si	N_2	S	Mn	O_2	H_2	N_2		Melting	Tapping		
A-3460	56.6	14.9	24.4	0.09	0.52	0.024	0.003	1.32	0.003	0.00041	0.012	2	17	42	3270	1
A-3461	58.3	14.8	25.4	0.10	0.56	0.029	0.006	0.18	0.006	0.00025	0.023	5	22	127	3100	1
A-3462	58.8	15.4	24.7	0.10	0.59	0.028	0.008	0.34	0.003	0.00028	0.023	3	47	233	3250	1
A-3464	56.5	15.3	27.1	0.07	0.62	0.022	0.007	0.02	0.002	0.00021	0.020	2	108	21	3440	2
A-3465	54.2	15.0	27.5	0.04	0.62	0.022	0.005	0.26	0.005	0.00019	0.014	4	95	24	3400	2
A-3466	63.0	13.2	22.4	0.04	0.04	0.007	0.006	Nil	0.153	0.00022	0.008	3	18	25	3300	2
A-3831**	59.8	14.9	24.0	0.06	0.70	0.027	0.005	0.04	0.012	0.00012	0.018	3	15	97	3250	1
A-3832**	54.0	16.2	27.6	0.08	0.57	0.030	0.003	0.07	0.019	0.00016	0.024	5	6	5	3240	1

* Melting Procedure No. 1. Alloy held molten 10 minutes and then tapped.

Melting Procedure No. 2. Alloy held molten until pressure dropped to approximately 20 microns and then tapped.

** Results of zirconium analysis will be reported later.

Note 1. All castings were removed from the molds at about 600°F., placed in a furnace at 1200°F., allowed to reach temperature, and furnace cooled.

Note 2. All of the heats were poured into copper mold No. 10 which, before each cast, was coated with aluminum paint and heated to 800°F.

oxygen content (during melting), as evidenced by the high oxygen in the finished melt. Another heat of this type will be made in an attempt to obtain a low oxygen content so that this heat may be compared with heats of low oxygen contents previously made using regular melting stock. This comparison should give an indication of the effect of minor impurities in the melting stock on the properties of this alloy.

The oxygen contents of the six heats correlated well with the cleanliness of the castings as determined metallographically. The casting from Heat A-3466, with high oxygen content, was very dirty, while the castings from Heats A-3460, A-3462, A-3464, and A-3465, having low oxygen contents, were very clean.

The recovery of manganese was greater in Heat A-3460 than in the other heats. Maximum recovery of manganese appears to depend on the method of addition, and on the temperature at which and the time interval during which the alloy is held molten.

Since analysis for the boron in Heats A-3448 and A-3449 is extremely difficult and the stress-rupture properties of these two heats are not promising, these boron determinations have not been completed.

To get an indication of the grain size in the center of the wing sections and still obtain two stress-rupture bars from each casting, the central sprue or feeder of each casting was examined in an area where the grain structure should be similar to that in the wing sections. The area selected was in the center of the sprue parallel to the vertical axis, and 3/8 inch from the base. The ASTM grain size for each casting is recorded in Table 170.

Fifteen minutes after tapping, the castings from Heats A-3458 to A-3466, inclusive, were removed from the mold at about 600°F., placed in a furnace and treated at 1200°F., and furnace cooled. This has been standard practice for all castings during the past several months. Failure of the thermocouple during the heat treatment of the casting from Heat A-3464 allowed it to attain an unknown temperature. Metallographic examination of this casting indicated that the casting had been heated above 1600°F. Nevertheless, this heat will be tested in stress rupture.

Heats Made During September

The objective in melting the 60Cr-15Fe-25Mo type alloys during the month of September was to ascertain whether or not additions of zirconium or selenium, with and without manganese, would decrease the fragility of this alloy. It is thought that even though the sulphur content of the alloys is below 0.01 per cent, a brittle, intercrystalline sulphide film may form. The addition of manganese, zirconium, or selenium may change the form of the sulphide inclusions to a less harmful, globular one and may lead to a more random dispersion of the inclusions. Albert Gagnebin (August, 1947, issue of "American Foundryman") stated that "selenium has the specified ability to coalesce the intergranular sulphides in cast steel and thereby improve its ductility". Selenium appears to combine with the sulphides in steel and promote their rejection from solution before solidification is complete. In addition, zirconium may tend to give a finer grained casting and to tie up nitrogen as a zirconium nitride. Manganese, in addition to combining with sulphur, may add some toughness to the alloys.

Eight 60Cr-15Fe-25Mo type alloys containing zirconium or selenium, with and without manganese, were melted and cast in the vacuum apparatus. The following table records the heat numbers, the amounts of alloy added, and when added:

Heat No.	% Mn	% Zr	% Se	Mn Added to
A-3831	-	0.10	-	-
A-3832	-	0.20	-	-
A-3833	-	-	0.10	-
A-3834	1.0	-	0.10	Charge
A-3835	1.0	-	0.10	Melt
A-3836	-	-	0.20	-
A-3837	1.0	-	0.20	Melt
A-3838	1.0	0.20	-	Melt

Manganese was added as fused metal, zirconium as a Si-Zr alloy (40.0 per cent Zr), and selenium as an Fe-Se alloy (56.8 per cent Se). The Si-Zr and Fe-Se alloys were added to the surface of the melts four minutes before tapping. When manganese was added to the melt, it was used in combination with either the zirconium or selenium alloy. The chemical analyses and grain sizes of Heats A-3831 and A-3832 are included in Table 170. The data for the remaining six heats will be included in next month's report.

Stress-Rupture Properties

Stress-rupture tests on several chromium-base alloy heats are in progress. These are described in the preceding section on "Engineering Properties of Heat-Resisting Alloys".

Future Work

Plans for the immediate future include:

1. Making of additional vacuum melts of the more promising alloys.
2. Modification of the composition of the 60Cr-15Fe-25Mo alloy to determine whether or not different ratios of chromium, iron, and molybdenum will lead to greater toughness in this type of material without too great a loss of stress-rupture properties.
3. Investigation of the effects of minor additions of alloys such as zirconium, manganese, aluminum, silicon, titanium, or selenium on the toughness and stress-rupture properties of the chromium-iron-molybdenum alloy.
4. Tests of alloys cast in both copper and refractory or sand molds to demonstrate the effect of freezing rate upon crystal structure of castings, and in turn its effect upon stress-rupture properties.

INVESTIGATION OF THE FUNDAMENTAL FACTORS PROMOTING HIGH-TEMPERATURE STRENGTH OF ALLOYS

(Work done by A. R. Elsea, A. B. Westerman, G. K. Manning, J. R. Doig, M. W. Mallett, and C. M. Schwartz)

Experimental Work

Cobalt-Chromium Binary Alloys

During the past month, work on this investigation consisted of melting, heat treating, and examining the microstructures of cobalt-chromium binary alloys for the purpose of checking the accuracy of the tentative equilibrium diagram.

In order to minimize nitrogen pickup during melting and heat treating, these operations were carried out in an atmosphere of purified argon. High-purity argon (99.6 per cent argon) was passed through magnesium perchlorate to remove most of the moisture. The partially dried gas was passed through copper coils immersed in a bath of acetone and carbon dioxide ice to reduce further the moisture content to a dew point of -80°C. The dried argon was then passed over titanium metal granules heated to 750°C. to remove nitrogen and oxygen. Samples of cobalt-chromium binary alloys melted and heat treated in this purified argon atmosphere contained from 0.005 to 0.009 per cent nitrogen.

A total of twenty-five cobalt-chromium binary alloys with chromium contents ranging from 41 to [illegible] per cent were melted using the argon protective atmosphere. The melting operations were carried out in an electric resistance furnace with a tubular Globar element. The highest

temperature attainable in this furnace (at the expense of a greatly reduced life for the heating element) is approximately 1700°C.; thus, it was impractical to attempt melting alloys containing over 73 to 75 per cent chromium because of their high melting points.

The first fourteen alloys melted were furnace cooled from the melting temperature to the temperature of the desired aging treatment. When the resulting ingots were sectioned, it was found that two, and sometimes three, separate and distinct layers existed. Chemical analyses of these separate layers showed a difference between top and bottom layers of as much as 34 per cent chromium (top-63.7 per cent chromium; bottom-29.3 per cent chromium). At first this separation was thought to be the result of immiscibility in the liquid state. However, it was later proved to be caused by selective segregation since this phenomenon occurred only when the alloys were furnace cooled through the solidification range. Alloys that were quenched from the liquid state were found to be quite homogeneous.

A series of eight cobalt-chromium alloys containing from 41 to 55 per cent chromium were melted in the purified argon atmosphere and then quenched in water to cause rapid solidification. These alloys are being examined metallographically to determine the composition of the eutectic.

A series of aging treatments on alloys containing from 41 to 73 per cent chromium have been carried out at temperatures ranging from 1150 to 1480°C. These temperatures and compositions have been selected so that critical points on the diagram can be checked by means of the Lever Law. Metallographic examination of these specimens has not been completed.

Effect of Nitrogen on Transformation Temperature of Cobalt

The anomalous differences reported in the literature for the crystallographic transformation temperatures of cobalt metal and of cobalt-chromium binary alloys were re-examined. It was considered likely that differences in nitrogen solubility might be responsible for much of the discrepancy in reported transformation temperatures, although no nitrogen analyses were reported in any case.

An experiment was planned in which the transformation temperature of a single sample of vacuum-cast electrolytic cobalt could be measured as a function of nitrogen content of the sample. Nitrogen would be added at various temperatures by contact with an atmosphere of molecular nitrogen and the transformation temperature of the cobalt would be determined in situ by a method such as measurement of the electrical resistance.

It was considered necessary first to determine whether or not nitrogen could be sorbed by the solid cobalt from atmospheres of molecular nitrogen at ordinary temperatures such as 900°C. For this determination, a sample of specially cleaned cobalt filings was prepared and the nitrogen sorption was measured at 900 and 1000°C. in a Sieverts apparatus(1). Within the limits of experimental errors (±0.002% by weight), no sorption was detected. In fact, slightly negative values (0.001-0.002%) were obtained, probably because of the evolution of a small amount (0.15 cc.) of residual hydrogen from the cobalt metal.

(1) A. Sieverts, "The Absorption of Gases by Metals", Z. f. Metallkunde, V. 21, 1929, pp. 37-46.

This was in agreement with Sieverts' failure to find any solubility of nitrogen in cobalt.(1) Accordingly, the attempt to change the nitrogen content by contact with molecular nitrogen was abandoned.

However, experiments to determine the effect of extremely small and extremely large nitrogen contents on the transformation of cobalt are being considered. Samples will be prepared from cobalt melted under purified argon and under cracked ammonia to obtain a wide variance in nitrogen content. Then the transformation temperatures of these samples will be determined probably by electrical resistance measurements.

Future Work

The experimental work associated with checking the present working diagram for the cobalt-chromium binary system will be completed.

The study of the effect of nitrogen on the transformation temperature of cobalt metal will be continued.

FUNDAMENTAL INVESTIGATION OF THE CAUSES OF CRACKING IN WELDS AND ADJACENT PARENT METAL

(Work done by C. B. Voldrich, R. D. Williams, and J. L. Foster)

Introduction

Microscopic examination of previously prepared sections of Timken alloy circular-groove specimens welded with 5/32-inch-diameter Types 312 (29Cr-9Ni), 430 (16Cr), 349 (19-9 W-Mo), and 330 (15Cr-35Ni) electrodes was continued. Additional examinations were made on Timken alloy-Vitallium wheel-and-bucket replicas welded with Types 316 (18Cr-12Ni-2Mo) and 349 electrodes.

(1) A. Sieverts and H. Hagen, Z. f. Physikal. Chem. (1934), 169A, p. 237.

In the study of the segregation observed in some of the above specimens, two samples containing lines of segregation were subjected to successive heat treatments at temperatures ranging from 700 to 1200°F., in 100°F. increments, and examined at the same locations after each treatment.

In connection with the study of the possible effects of sulphur and phosphorus on weld-metal cracking, another low-sulphur, low-phosphorus heat of Type 316 core wire was made to replace the unacceptable heat made last month.

Experimental Work

Macroscopic Study of Segregation in Weld Metal

Further Examination of Samples Welded in July. The results of examination of sections from four Timken alloy circular-groove specimens welded in July were described in the reports dated August 16, 1947, and September 16, 1947. These specimens were:

Specimen No.	5/32-Inch-Diameter Electrode Used
6	312 (29Cr-9Ni)
7	430 (16Cr)
8	349 (19-9 W-Mo)
9	330 (15Cr-35Ni)

A third section of each specimen, parallel to sections "j" and "k" (Figure 109, report dated August 16, 1947) and 1/32" below "k", was

examined this month. The observations made are given in Table 171. None of these observations furnished evidence of any definite relationship between the interblock cracking and the segregation in the weld. Further examination of these sections appears unwarranted at this time.

Examination of Early Timken Alloy-Vitallium Wheel-and-Bucket Replicas Welded With Types 316 and 349 Electrodes. Additional sections of the Timken alloy-Vitallium wheel-and-bucket replicas welded with Types 316 and 349 electrodes described in the report dated September 16, 1947, (pp. 877-880, and Figure 111) were examined metallographically. These sections were 1/32 inch below those studied last month.

Examination of the longitudinal and transverse sections of the replica welded with Type 316 electrodes revealed no new microstructural features. Segregation, similar to that reported last month, was observed both uniformly distributed throughout the weld and concentrated near the block extension cracks. A few wisps of segregation extended into the weld from the base metal.

In the transverse section of the specimen welded with Type 349 electrodes, no segregation was observed. This observation differed from that made on the previous section in that a few wisps of segregation had been found in the latter.

Examination of New Timken Alloy-Vitallium Wheel-and-Bucket Replica Welded With Type 316 Electrodes. A second section, "u", of the Timken alloy-Vitallium wheel-and-bucket replica welded last month with Type 316 electrodes (p. 880 and Figure 111B) was examined. On one side of the assembly only the root pass was laid; on the other side all four passes

TABLE 171. MICROSCOPIC EXAMINATION OF TIMKEN ALLOY CIRCULAR-GROOVE SPECIMENS WELDED WITH TYPES 312, 430, 349, AND 330 ELECTRODES

Specimen	Section(1)	Observations(2)
6	1	No surface cracks evident. Small junction crack starting under top surface of the weld. A few small fine lines of segregation. Very small areas of delta ferrite.
7	1	Discontinuous and irregular crack started under weld surface and extended to junction. No evidence of segregation. Fine, uniformly distributed delta ferrite. One apparent line of segregation observed in section "k" now appeared as part of the crack.
8	1	Crack, originating at block junction, extended about 2/3 distance across the weld. No segregation. Delta ferrite uniformly distributed in weld.
9	1	Junction crack extended to surface. Grain distortion associated with crack. Lines of segregation adjacent to crack; some randomly distributed in weld.

(1) For location and orientation of surface "1", see Figure 109 of the report dated August 16, 1947.

(2) Magnifications of 100X and 1000X used. Whenever segregation is mentioned, it refers to fine nodules occurring in stringers at the grain boundaries. Typical stringers of segregation are shown in Figure 110 of the report dated August 16, 1947.

were deposited. Section "u" was taken parallel to and 1/32 inch below section "t". The observations made on section "u" are given in Table 172. In section "t" no interbucket junction cracks were observed, but in section "u" interbucket junction cracks were found at Junction 1 in both the single-pass and multipass welds. Although no evidence of the relationship between weld-metal structure and interbucket extension cracking has yet been found, one more section of this specimen will be examined.

Study of Segregation. While small wisps of segregation have been found in welds in both Timken alloy circular-groove specimens and Timken alloy-Vitallium wheel-and-bucket replica specimens, no definite identification of this segregation has been made, although it is suspected that it consists of one or more complex carbides. Examination at high magnification has already shown that those apparent lines or wisps of segregation are actually strings of very minute nodules.

Some clue to the identification of these small lines of segregation might be obtained if specimens with definitely located lines of segregation were treated so as to precipitate more of the segregating constituents and thus provide sufficient amounts of the material for identification by metallographic and X-ray diffraction methods. It is evident that, until the segregate and the material precipitated by heat treatment are identified, there is no certainty that the precipitate and the segregate are the same. It was decided, therefore, to heat treat specimens for 24 hours at successively higher temperatures, starting with 700°F. and increasing in 100°F. increments, and to examine them metallographically after treatment at each temperature.

TABLE 172. MICROSCOPIC EXAMINATION OF TIMKEN ALLOY-VITALLIUM WHEEL-AND-BUCKET REPLICA WELDED WITH TYPE 316 ELECTRODES

Specimen Number and Junction	Section	Observations
10-Junction 1	u	Small junction crack on single-pass side, larger one on multipass side. Few small lines of segregation on single-pass side, more in first pass on multipass side.
10-Junction 2	u	No junction crack. Structure similar to that in Junction 1.
10-Junction 3	-	(1)
10-Junction 4	u	No junction crack. Structure similar to that in Junction 1.
10-Junction 5	u	No junction crack. Structure similar to that in Junction 1.

(1) Used in study of segregation discussed later in this report.

Note: A few lines of segregation originating in the Vitallium were again observed in the weld.

Two samples were selected for this study - one from a Timken alloy circular-groove specimen welded with Type 316 electrodes, and the other (Specimen 10-Junction 3) from the new Timken alloy-Vitallium wheel-and-bucket replica also welded with Type 316 electrodes. These were examined as welded; small areas including wisps of segregation were delineated with a diamond micromarker, and these were photographed. The samples were then sealed in evacuated pyrex glass tubes and heat treated at 700°F. Since it was found that the markings made with the micromarker did not remain visible after the light buffing and re-etching necessary subsequent to each heat treatment, the areas of interest were relocated by scratching the specimens with a needle, and the specimens were polished lightly, etched, and photographed. This cycle was then repeated; each time the next higher temperature was used. At the higher heat-treating temperatures, the specimens were sealed in evacuated quartz tubes for protection from the furnace atmosphere.

Microscopic examination between heat treatments revealed no structural changes in either specimen until after the 1200°F. treatment. In the circular-groove specimen, sufficient precipitate was present after treatment at 1200°F. to outline the grain boundaries of the weld metal. Thus, it was apparent that what had been considered a line of segregation in the as-welded specimen was merely a portion of a grain boundary which was particularly evident because of more precipitation in that location during solidification than in the other portions of this grain boundary. No further work will be done on the circular-groove specimen.

After the 1200°F. heat treatment, the wheel-and-bucket replica specimen revealed a precipitate in the vicinity of the original line of segregation. Examination under polarized light indicated that numerous silicate inclusions were present in the weld metal. One or more constituents which had the appearance of carbides were also visible, but positive identification of the precipitate or of the original segregate could not be made.

This study will be continued by heating the wheel-and-bucket replica specimen at temperatures above 1200°F. If the amount of segregation continues to increase, efforts will be made to identify it by X-ray diffraction.

Effect of Sulphur and Phosphorus on Weld-Metal Cracking

In the reports of June 16, 1947, through September 16, 1947, the possible relationship between sulphur and phosphorus and weld-metal cracking was discussed, and plans were described for making test materials low in these two elements. A satisfactory electrode coating and Timken alloy heat have been made, but the Type 316 core wire heat was found to be excessively high in nitrogen. Another Type 316 core wire heat was, therefore, made in September. The analysis of this heat (A-3886) was as follows:

Element	Per Cent
Carbon	0.02
Manganese	1.88
Silicon	0.56
Sulphur	0.013
Phosphorus	0.005
Chromium	17.8
Nickel	13.8
Molybdenum	2.37
Nitrogen	0.114

The sulphur content of this heat was so high that the heat had to be rejected. The only difference between the materials used for this heat and those used for the last was that another lot of electrolytic iron, made to the same specification, had been used. Upon subsequent analysis, it was found that this iron had a sulphur content of 0.008% instead of the 0.003% found in the first lot. A new lot of electrolytic iron will be ordered as soon as test samples, submitted by the producer, indicate that the material will be acceptable.

While the nitrogen was still higher than desired, it is believed that it would have been acceptable for the tests, because of the low nitrogen of the Timken alloy heat. However, a further reduction in nitrogen will be sought in the next core wire heat.

Future Work

1. Heat treatment of the Timken alloy-Vitallium wheel-and-bucket replica welded with Type 316 electrodes and containing lines of segregation will be continued in an effort to identify the segregating constituents.
2. Another section of the Timken alloy-Vitallium wheel-and-bucket replica recently welded with Type 316 electrodes will be examined microscopically.
3. Another low-sulphur, low-phosphorus heat of Type 316 core wire will be made. Further precautions will be taken to reduce the nitrogen content below that in the two heats made thus far.

4. None of the tests made throughout this investigation of the fundamental causes of cracking in welds and adjacent parent metal has provided definite evidence of the relationship between the composition and structures of the metals involved and the weld-metal cracking observed. There is no certainty that the tests now in progress will provide this fundamental information.

Further methods of attack may, and probably will, be necessary. Among these is the long-needed development of more information on some of the fundamental properties of the metals involved. The studies of the constitution of some of these metals, now in progress, have not reached a stage where the information can be applied to the thermal and chemical changes occurring during welding. However, certain other physical properties of metals could be studied with currently available equipment, and it is quite possible that some fundamental information on the causes of weld-metal cracking might be obtained. For example, a study of the expansion and contraction properties of the high-temperature alloys involved, at temperatures in excess of 2000°F., might prove fruitful. Similarly, a measurement of their tensile strengths between 2000°F. and their melting points might provide useful information.

A test program covering the investigation of some of these properties is now under consideration and it is expected that the initial line of attack will be selected fairly soon.

FUNDAMENTAL STUDIES OF CERAMIC MATERIALS

(Work done by G. R. Eusner, H. Z. Schofield, and C. R. Austin)

Experimental Work

Effect of Burning-Schedule Variations on Properties of Alumina

The determination of the relationship between the thermal history and the physical properties of burned alumina bodies was continued. In this work, a series of No. 38900 Alundum[1] specimens which were formed by dust pressing in the usual manner at a pressure of 5000 pounds per square inch, were burned at temperatures ranging from 1000 to 3300°F.,[2] in increments of about 250°F. The specimens were placed in the furnace, burned at the particular temperature of the test, and then furnace cooled; the details of specimen preparation were presented in the report dated September 16, 1947. The burning conditions and properties of the bodies are given in Table 173; the relationships between the burning temperature and the linear shrinkage, bulk density, and modulus of rupture of the specimens are also shown in Figures 114, 115, and 116, respectively.

Within the limits of this investigation, no critical temperature ranges were evident in which abrupt changes occurred in these particular properties of the No. 38900 Alundum body. In general, and as was anticipated, the linear shrinkage, bulk density, and modulus of rupture

(1) Obtained from Norton Company, Worcester, Massachusetts.

(2) Data obtained on specimens burned in the temperature range of 1000 to 2500°F., inclusive, were discussed in the report dated September 16, 1947, and are included in this report to facilitate a comprehensive evaluation of the body.

TABLE 173. EFFECT OF BURNING TEMPERATURE ON PROPERTIES OF BODIES OF NO. 38900 ALUNDUM

Burning Temp., °F.(1)	Weight Loss, Per Cent	Linear Shrinkage, Per Cent	Water Absorption, Per Cent	Bulk Density, Gm./cc.	Modulus of Rupture, Lbs. Per Sq. In.
1000(2)	8.5	No apparent change in length	-(3)	-(3)	-(3)
1250(2)	8.5	Ditto	-(3)	-(3)	-(3)
1500(2)	8.5	"	16.8	2.41	-(4)
1750(2)	8.5	"	16.8	2.37	-(4)
2000(2)	8.8	0.04	16.9	2.41	-(4)
2250(2)	8.8	0.3	16.9	2.41	-(4)
2500(2)	8.9	1.3	16.5	2.43	800
2750	8.7	4.1	12.0	2.70	5,350
3020	8.6	8.0	7.7	3.10	9,380
3100	8.3	10.0	5.3	3.29	12,710
3300	8.5	11.3	0.11	3.75	Specimens not suitable for this test

(1) Specimens were heated to the burning temperatures at a rate of approximately 125°F. per hour and soaked for 30 minutes at the indicated temperatures.

(2) Data obtained from report dated September 16, 1947.

(3) Specimens disintegrated in the water used in the absorption test.

(4) Specimens were too fragile for strength determinations.

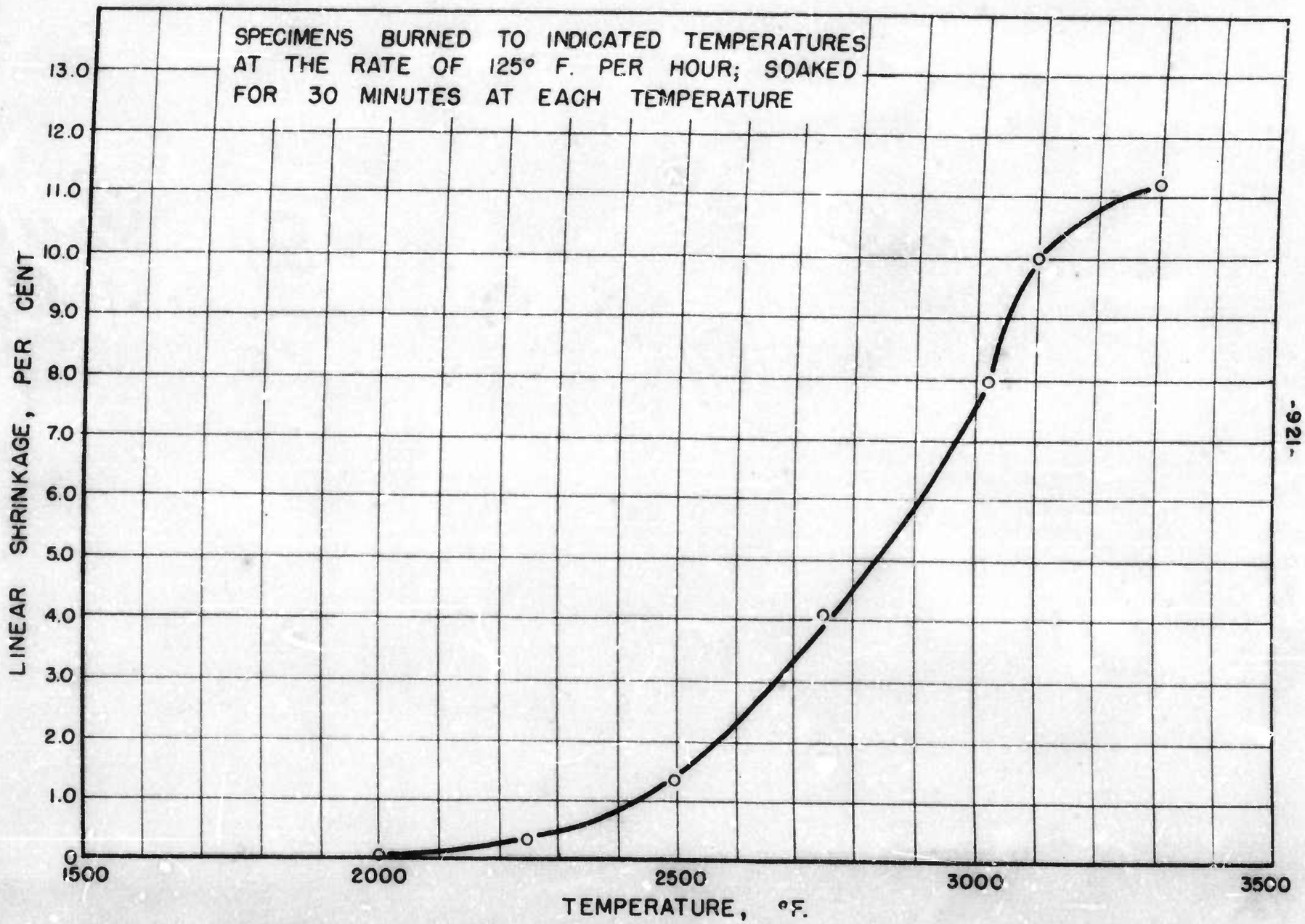

FIGURE 114. EFFECT OF BURNING TEMPERATURE ON LINEAR SHRINKAGE OF SPECIMENS OF NO. 38900 ALUNDUM

0-6470

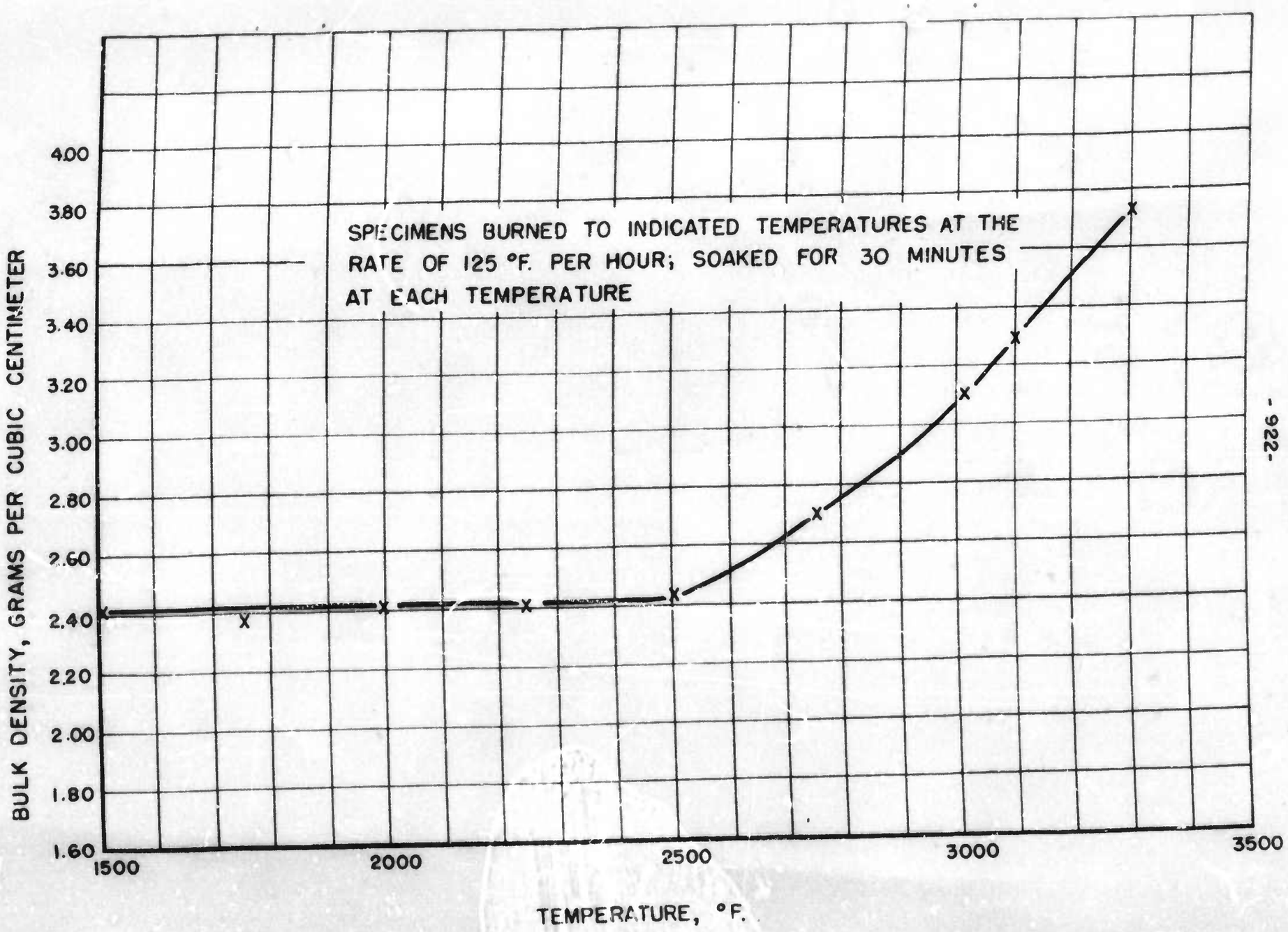

FIGURE 115. EFFECT OF BURNING TEMPERATURE ON BULK DENSITY OF SPECIMENS OF NO. 38900 ALUNDUM

0-6471

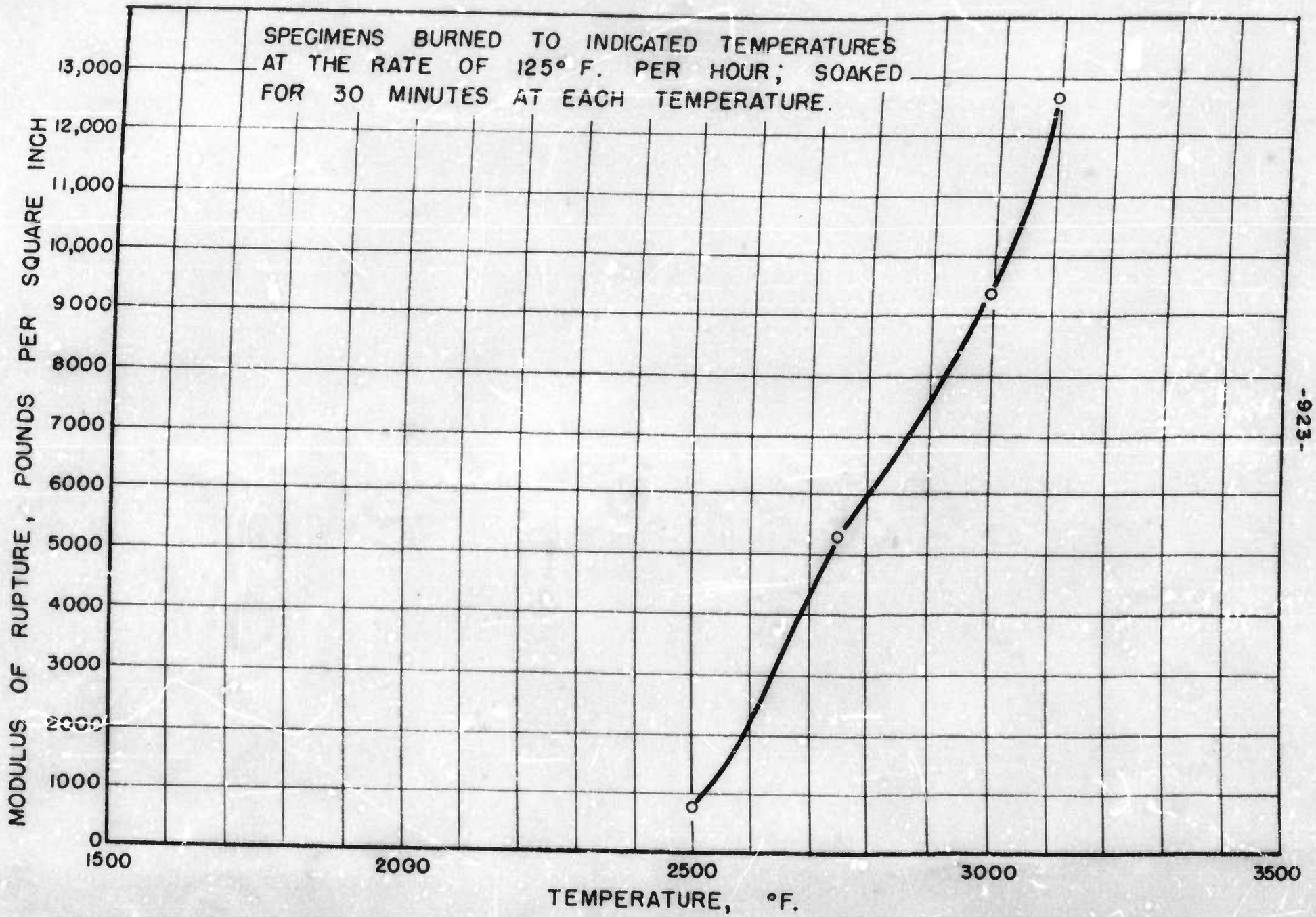

FIGURE 116. EFFECT OF BURNING TEMPERATURE ON MODULUS OF RUPTURE OF SPECIMENS OF NO. 38900 ALUNDUM

0-6472

of the body increased, and the water absorption decreased as the burning temperature was increased; these trends, which were uniform in pattern, were especially apparent in the specimens burned at temperatures of 2500°F. or higher. The loss in weight of the specimens during burning ranged from 8.3 to 8.9 per cent and did not appear to be influenced significantly by the burning temperature.

The specimens burned at temperatures of 2250°F. or lower were extremely fragile, and satisfactory strength determinations were not possible. The properties of this particular group of specimens were discussed in detail in the previous report.

Determination of Length Changes in No. 38900 Alundum During Burning

The work reported in the foregoing section indicated that changes in the properties of the No. 38900 Alundum body occurred at a relatively uniform rate, and that within the limits investigated, no critical temperature range was observed in which a change in the properties was sudden or abrupt. In order to obtain additional knowledge of the changes taking place during the burning operation, preliminary attempts were made to observe continuously the length changes occurring during the burning of a wax-bonded specimen of No. 38900 Alundum from the unburned to the burned condition. Two methods of observation were employed; these are described in the following paragraphs.

Thermal Expansion Apparatus. An unburned, wax-bonded specimen of No. 38900 Alundum was mounted in the usual manner in a differential-type, fused silica dilatometer, which is commonly used for thermal expansion determinations. The specimen was heated at a rate of 250°F. per hour from room temperature to 1820°F., the maximum operating temperature of the equipment, and was held at 1820°F. for two hours. The specimen was cooled in place and then reheated, using the same heating cycle, to a temperature of about 1700°F. to determine whether or not additional changes of an abrupt nature might occur in the specimen. The linear expansion data so obtained are given in Figure 117.

An abrupt expansion of 0.06 per cent and an equally abrupt contraction of approximately 0.05 per cent occurred over the temperature range of 55 to 175°F.; these changes probably resulted from melting of the wax binder and the release of the adhesive force of the binder. Over the temperature range of 800 to 875°F., a slight shrinkage of 0.02 per cent occurred, probably because in this range the binder was eliminated from the specimen. Over the range of 1700 to 1820°F., and during the two-hour period while the specimen was at 1820°F., a decrease in length of 0.05 per cent was observed; this was probably a result of the start of sintering of the body. Excepting for the temperature ranges discussed, the expansion of the body during heating from the unburned condition at room temperature to a temperature of 1700°F. occurred at a uniform rate.

During reheating of the specimens from approximately 300 to 1700°F., no abrupt changes in length were observed. The expansion of the body increased at a uniform rate throughout this part of the test.

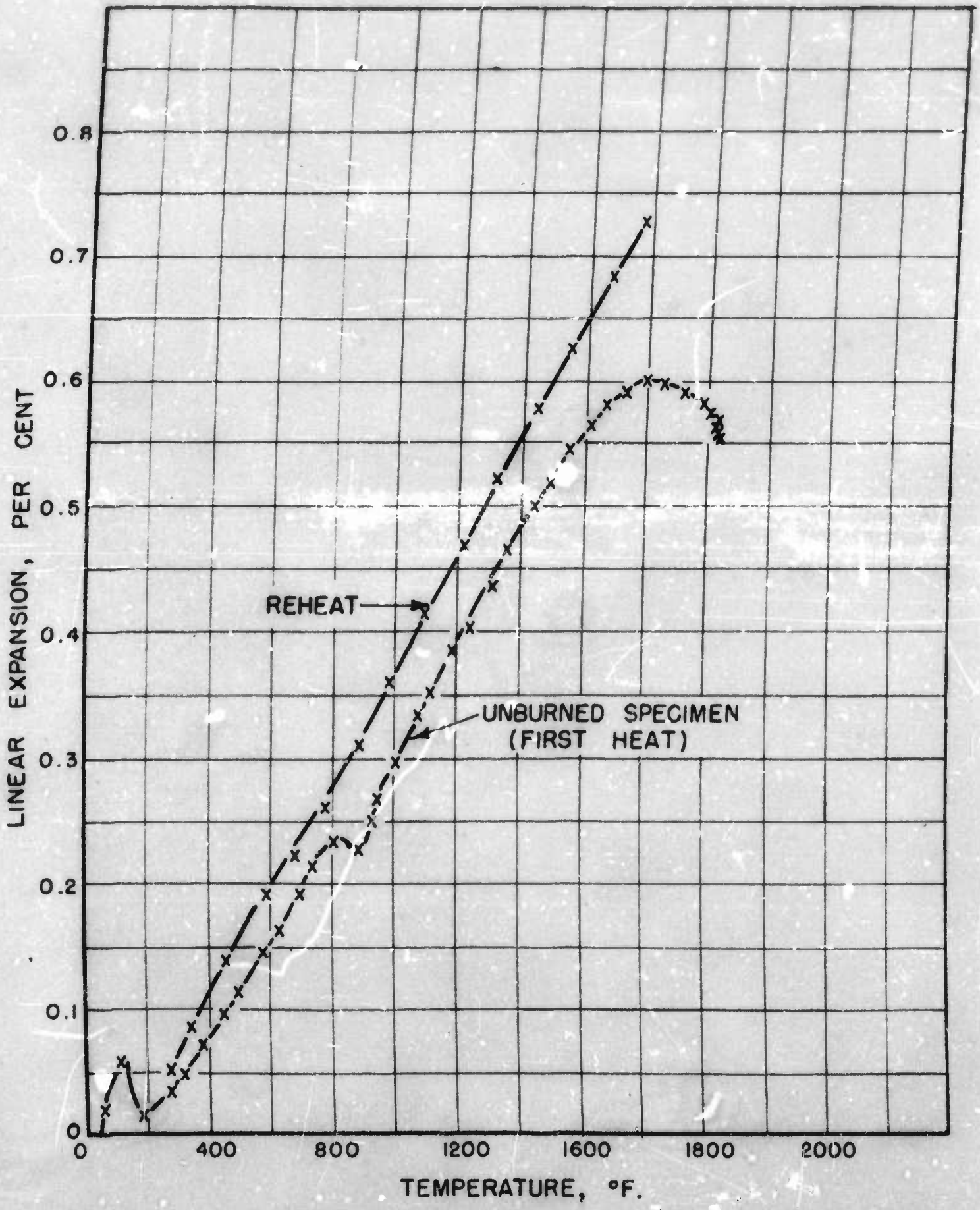

FIGURE 117. EXPANSION OF SPECIMEN OF NO. 38900 ALUNDUM DURING BURNING FROM ROOM TEMPERATURE TO 1820° F.

0-6473

Direct Optical Measurement. Attempts to observe the changes in the length of a No. 38900 Alundum specimen, during burning by means of a fused silica dilatometer necessarily were confined to relatively low temperatures, since the maximum operating temperature of the apparatus was approximately 1820°F. In an effort to observe the changes occurring at temperatures above 1820°F., preliminary attempts were made to take direct optical measurements by using modifications of a technique which reputedly was successful in the evaluation of fire-clay refractories(1).

Two slots, approximately 3/4" x 3/4", were cut about 6-1/4 inches apart in an Alundum tube, 1 inch in diameter and about 8-1/4 inches in length. Fused alumina grains were spread evenly on the inside bottom of the tube, which was supported horizontally, and a specimen of No. 38900 Alundum, 5/16" x 5/16" x 7-1/2", was placed on the grains so that the ends of the specimen were visible through the two slots in the tube. This assembly then was placed in a gas-fired furnace and was taken from room temperature to 3100°F. at a rate of 125°F. per hour. During heating, the specimen was observed by means of a Gaertner laboratory telescope having a minimum focal length of approximately 6 feet, to determine whether or not the outline of the specimen might be sufficiently sharp at the higher temperatures to permit accurate measurements of length. No actual measurements were made, however, during this preliminary evaluation of the technique. The arrangement of the specimen in the tube is illustrated in Figure 118.

(1) D. K. Stevens and R. E. Birch, "Shrinkage Rates in Firing Fire-Clay Refractories", Jour. Amer. Ceram. Soc., 30 (4), 109-113 (1947).

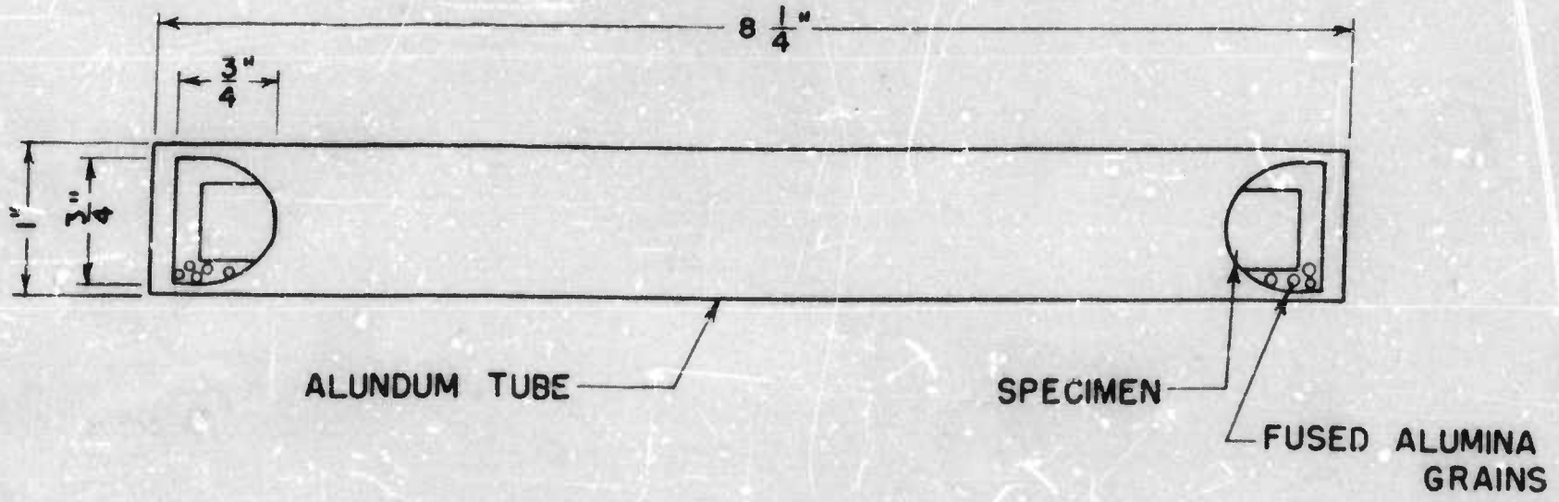

FIGURE 118. ARRANGEMENT FOR DIRECT OPTICAL MEASUREMENT OF LENGTH CHANGES IN SPECIMEN DURING BURNING

0-6474

At temperatures of about 2500°F. and above, the outline of the specimen became indistinct, and differentiation of the specimen from the mounting was not obtained because of the essentially "black body" conditions present in the furnace. Attempts will be made to overcome this condition by the use of appropriately colored filters in the telescope or by changing the background.

Tensile Testing

Work was continued on the determination of the tensile strength of a burned specimen of No. 38900 Alundum at elevated temperatures, and on the development of a more efficient means of forming tensile specimens. A specimen, 1" x 1" x 7", was formed in the usual manner by pressing, was preburned at 2500°F., and then was machined to the proper size and shape by using regular machine-shop techniques. Allowances were made for shrinkage of the specimen during burning. The machined specimen was packed in fused alumina grains in a refractory Alundum tube, was burned in a vertical position at 3100°F., and then was reburned at 3300°F. by using the normal burning techniques described previously.

The reburned specimen was straight and within dimensional tolerances. The adapters of the high-temperature tensile-testing apparatus fitted well to the ends of the specimens. The load was applied to the specimen at the rate of 2000 pounds per square inch per minute. The tensile strength of the specimen of No. 38900 Alundum, reburned at 3300°F., was 1500 pounds per square inch when tested at 1800°F. A very coarse crystalline structure was apparent throughout the specimen.

Future Work

Work will be continued on the determination of the changes which occur during the burning of specimens of No. 38900 Alundum.

A study will be made of crystal formation in alumina, beryllia, and zircon, and of the means of controlling crystal size in specimens of these bodies.

HCC:ma

October 24, 1947